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## A METHOD FOR THE QUANTITATIVE DETERMINATION OF SALICYLIC ACID IN THE PRESENCE OF ACETYL SALICYLIC ACID.

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**D**URING the past few months both state and federal laboratories have found many adulterated samples of acetyl salicylic acid (aspirin), one of the most common adulterants of this product being salicylic acid.

Among the qualitative tests for the differentiation of acetyl salicylic acid and salicylic acid is the test with bromine water. Bromine water gives a precipitate with salicylic acid, but does not give a precipitate with acetyl salicylic acid.<sup>1</sup> This fact and the method of Fr. Freyer<sup>2</sup> for determination of salicylic acid by means of a N/10 bromine solution were utilized for the quantitative determination of salicylic acid in mixtures of the two drugs. The method of Freyer is practically that of the U. S. P. for the determination of phenol. The end product, tri-brom-phenol, is the same with both drugs.

Two solutions were prepared, one containing 100 mg. of salicylic acid in 100 cc., and the other 100 mg. salicylic acid and 100 mg. acetyl salicylic acid in 100 cc. Ten cc. of the solution, equivalent to 10 mg. of U. S. P. salicylic acid, were used for a determination. Twenty cc. of N/10 bromine, U. S. P., were added, followed by 75 cc. water and 5 cc. hydrochloric acid. The mixture was allowed to stand, with occasional shaking, in a glass-stoppered flask for ten minutes, when 5 cc. of potassium iodide, T. S., were added, and the liberated iodines titrated with N/10 sodium thiosulphate, V. S. The two solutions required the same number of cc. of N/10 thiosulphate and showed the presence of 9.9881 mg. of salicylic acid in 10 cc. samples of each solution.

A 50 mg. sample of the same salicylic acid showed, by direct titration with N/10 KOH, a purity of 99.9 percent, which result checks very closely with the preceding.

It must be remembered, however, that a cold aqueous solution of acetyl salicylic acid decomposes slowly, forming salicylic acid, and if heated the reaction is comparatively rapid.

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1. Fuller, *Qualitative Analysis of Medicinal Preparations*, p. 26.

2. *Chem. Zeitung*, vol. 20, p. 820.

Therefore, determinations must be made at not above room temperature and soon after bringing the samples into solution. To illustrate: 10 cc. of the above mixture of salicylic acid and acetyl salicylic acid showed, after standing forty-eight hours, 11.53 mg. of salicylic acid, or an increase of 1.542 mg. during that length of time.

Six atoms of bromine correspond to one molecule of salicylic acid, or 1 cc. of the N/10 bromine consumed is equivalent to 0.002283 gm. of salicylic acid.

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